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PATENT SPECIFICATION

DRAWINGS ATTACHED

883,448



Date of Application and filing Complete Specification: July 30, 1959.

No. 26153/59.

Application made in United States of America on Aug 6, 1958.

Complete Specification Published: Nov. 29, 1961.

Index at acceptance:—Classes 2(6), P2A, P2C(8B:12A:16C:17:20B), P2D(1A:2A), P2K(7:8), P2T2A, P7A, P7C(8B:12A:16C:17:20B), P7(D1B:K2:T2A); 2(5), R14C(6:10:11:12), R32C(6:10:11:12:); and 70, Q5X.

International Classification:—C08d, C08c, g.

COMPLETE SPECIFICATION

Microbicidal Elastomer Articles

5 We, THE D. S. BROWN COMPANY, a corporation organized and existing under the laws of the State of Ohio, United States of America, of East Cherry Street, North Baltimore, State of Ohio, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

10 This invention relates to microbicidal elastomer articles and their manufacture, and particularly, to articles for contact with milk.

15 Modern dairy practice involves the use of milking machines which must be maintained as free from contamination as possible. The milking machines include a set of elastomer inflations or teat cup liners, which are commonly molded from natural or synthetic rubber. The milking machines, including the inflations, are washed between milkings, and a sterilizing agent may be used. However, washing and sterilizing methods vary widely. At times, the equipment is thoroughly washed 25 twice a day, but very frequently, at least one wash consists only of a cold water rinse. At times, it has been found that rinsing after a milking is omitted.

30 Since milk furnishes an excellent nutrient media for bacteria, the maintenance of the most sanitary conditions is highly desirable. Prior studies have shown that the rubber inflations harbor bacteria, which come from the skin of the animal or may come from infected animals, or are picked up in handling and when not in use. The bacteria contaminate the milk, and may also infect the animals. The situation is improved by using synthetic rubber inflations, which build up lower bacterial concentrations than natural rubber. However, the presence of bacteria is a problem whatever type of inflation be employed, and the prior cleaning and sterilizing procedures are not as effective as desired. The

situation is especially aggravated by improper cleaning procedures. 45

It is therefore a major object of the present invention to provide microbicidal milking machine parts, especially inflations, which combat these hazardous tendencies. 50

A particular object is to provide milking machine inflations which are bactericidal, or bacteriostatic, to at least prevent bacterial growth, and preferably, reduce the bacterial count on the surface and in the interstices of the inflations. The invention acts to maintain the sterility of the milk, and to prevent infection of the animals by contaminated inflations. In particular, mastitis in herds is combated. The invention is also useful in other applications where milk is in contact with elastomeric articles, especially in handling and processing milk, as in rubber tubing, gaskets, liners, and other elastomeric articles which are contacted with milk. The margin of human error in the various applications is significantly reduced. 55 60 65

A further object is to provide bactericidal or bacteria-resistant elastomer articles which are active against the organisms encountered in handling milk and which articles retain their activity over repeated use. 70

Another object is to provide a microbicidal elastomer article from which the microbicidal agent is not substantially removed by water or milk. 75

Additional objects are to provide a microbicidal elastomer articles for contact with milk, a method of manufacture, and improvements in methods of handling milk and in dairy operations and the like. These and other objects, advantages and functions of the invention will be apparent on reference to the specification taken in conjunction with the attached drawing, in which: 80 85

Figure 1 is a side elevational and partly longitudinal sectional view of a microbicidal milking machine inflation according to the

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invention, having the same shape and appearance as a conventional inflation, and

Figure 2 is a top plan view of the inflation.

5 The invention provides a cured micro-
bicidal elastomer article employed in contact
with milk and having dispersed therethrough
a microbicidal agent. A preferred embodiment
10 constitutes a milking machine inflation, as
illustrated in the drawings. The microbicidal
agent is intimately and substantially homo-
geneously dispersed throughout the inflation,
such as the illustrative inflation 1, so that it
15 does not appear as a separate element of the
article, but is at least an extremely finely
divided component thereof.

The illustrative embodiment shows one
conventional inflation structure 1, but any of
the several structures adapted for the several
20 types of milking machines may be employed
as well. The inflation is constructed to pro-
vide an enlarged cup or chamber 2 adjacent
the top, for insertion of the teat. At the
opposite end 3, the milk is removed by means
25 of a vacuum connection, not shown. A steel
shell or cup (not shown) is emplaced around
the upper wide portion 4 of the inflation. A
vacuum connection is provided in the shell,
to perform the milking operation involving
30 alternate expansions and contractions of the
inflation in this area.

The invention also includes a method of
manufacturing a microbicidal elastomer article
which includes intimately incorporating a
microbicidal agent in a raw elastomer mix,
35 molding the resulting mix in the desired
shape, and curing the molded mix. Thus,
a microbicidal agent effective against the
microorganisms associated with milk is dis-
persed substantially homogeneously by mix-
40 ing, with raw rubber, plasticizers, fillers and
other conventional additives. The resulting
mix is molded in the shape of the inflation,
and the molded mix is cured. The inflation
is then ready for repeated use in a milking
45 machine. Improvements are likewise pro-
vided in other applications involving contact
of milk with elastomer surfaces.

The invention contemplates the use of any
microbicidal agent effective against the harm-
50 ful or pathogenic microorganisms encoun-
tered in handling milk, which is preferably
not more than slowly soluble in water and
milk when present in the elastomer article.
Phenolic microbicidal agents are very advan-
55 tageously employed, especially those having
but limited solubility in water. They may
be employed as such or in the form of their
salts. It is advantageous to employ the
phenols as such, since their water solubility
60 is reduced and they may be distributed very
uniformly in the article due to their increased
solubility in the organic components, such as
the plasticizers. It also appears that the
articles resulting from use of the phenols
65 *per se* are more effective bactericidally. Other

agents are contemplated, such as silver salts,
e.g., silver chloride. The microbicidal agent
should of course be nontoxic under the con-
ditions of use and in the amounts employed.

Only a minor effective amount of the bac-
70 tericide is incorporated in the elastomer
article, sufficient to inhibit bacterial growth
thereon, preferably 0.25% to 20% by weight
based upon the raw elastomer content of the
75 article. A minimum proportion of about
2% is further preferred. The higher the
concentration, the more rapid and complete
is the kill of bacteria. Consequently, the
preferred upper value may be exceeded in
80 appropriate circumstances to further increase
the effectiveness, provided that the concen-
tration is not so high as to prevent the curing
and formation of a satisfactory article.

The proportion is selected so as to not
unduly interfere with curing or vulcanizing
85 the molded mix in the shape or form of the
desired article. The curing of natural rubber
is apparently affected more by higher pro-
portions than the curing of the synthetic
rubbers. Some increase in curing time is
90 tolerable in view of the results provided by
the bactericidal article.

Milking machine inflations according to
the invention have undergone a number of
95 tests which have demonstrated their effective-
ness. Several of these tests are summarized
in the examples which follow, for purposes
of illustration. The results showed that the
inflations successfully combatted the bacteria
commonly encountered, to furnish increased
100 safety and guard against contamination of
milk and of the animals. The results were
especially good with the preferred neoprene
inflations, and the utility of other synthetics
and natural rubber was demonstrated. The
105 invention thus contemplates articles con-
structed of suitable elastomers including
natural rubber and synthetic elastomers such
as neoprene, butyl rubber, Buna (Registered
Trade Mark) S, Buna N, Thiokol (Registered
110 Trade Mark) rubberlike polymers, and poly-
urethane.

The following examples are furnished to
provide a more complete understanding of
the invention, but it is to be understood that
115 the invention is not limited to the compo-
nents, proportions and conditions set forth
therein, which are only illustrative.

The inflations used in the following and
subsequent examples were produced by in-
120 corporating the bactericidal agent in the raw
rubber mix, followed by molding and curing
or vulcanizing. Thus, the raw rubber solid
was masticated in a mill or mixed until plastic.
Then, the bactericide, and conventional plas-
125 ticizers, fillers, and accelerators were added,
followed by mixing for 3—15 minutes. The
proportions of the bactericide were calcu-
lated in percentages by weight, based on the
amount of raw rubber solid employed. An 130

oil-soluble bactericide, such as o-phenylphenol, may be dissolved or dispersed in the plasticizer for incorporation in the mix. The agent may be melted and the liquid mixed with the plasticizer, or agent in solid form may be intimately dispersed. This compounded rubber stock is molded in the shape or form of an inflation and then cured in the usual manner, e.g., with steam at 287° F. under pressure, for 20–30 minutes.

EXAMPLE 1

Two standard types of neoprene (G) (a sulphur modified chloroprene containing about 0.75% of sulphur) inflations containing from 0.25% to 2% of sodium o-phenylphenol were tested for bactericidal activity against *Escherichia coli*, *Staphylococcus aureus*, and a *Pseudomonas* species isolated from a mastitis case, in suspension in physiological saline. These organisms are commonly found in milk. *E. coli* is representative of intestinal disorders which are carried into milk. *Pseudomonas* is a very troublesome organism found in mastitis cases and difficult to eliminate. Any reduction of the organisms is considered quite beneficial.

The inflations were filled with the saline suspensions of the individual organisms and allowed to stand under atmospheric conditions. High concentrations of the organisms were used, to provide severe tests. Samples of the liquid were removed from the inflations after standing for periods of time, plated, incubated, and the colonies of bacteria counted.

At 0.25% of bactericide, progressive reduction of the bacteria count resulted over a 24-hour period, from 6 million per ml. to less than 10 thousand for *E. coli*, and from 9.4 million to 1.5 million for *S. aureus*. The *Pseudomonas* count was reduced but not significantly.

At 0.5% of bactericide, the results were comparable for *E. coli* and considerably better for *S. aureus*, from 9.4 million to 1.4 million in one hour, and to less than 10 thousand at 3 and 24 hours. *Pseudomonas* reduced from 5.2 million to 2 million in 24 hours.

At 2% of bactericide, the results were improved for *E. coli*, the count being reduced to

400 thousand in one type inflation and less than 10 thousand in the other at 3 hours, both being less than 10 thousand at 24 hours. *Pseudomonas* reduced from 5.2 million to about 3 million in 24 hours.

EXAMPLE 2

Inflations having various quantities of either o-phenylphenol or sodium o-phenylphenol were tested for bactericidal activity in the manner of Example 1 against a *Pseudomonas* species and *Staph. aureus*. The *Pseudomonas* suspension in saline had a count of 1.75 million per ml. at the start and the control (in a glass container) a count of 2 million at 24 hours. The *Staph.* suspension in saline had a count of 1.2 million at the start and the control a count of 4.4 million at 24 hours, indicating multiplication on cellular debris.

Neoprene (G) inflations containing 0.25% to 5% o-phenylphenol reduced the *Pseudomonas* count to 0.1 to 0.2 of the initial count in 3–4 hours, and the counts were further reduced in 24 hours, to 27 thousand in the case of 5% bactericide. Multiplication of the *Staph.* took place in 1 hour, and reduction had commenced in 3–4 hours. At 24 hours, the count was very low in each case, 75 thousand or lower.

Neoprene (G) inflations containing 2% and 5% sodium o-phenylphenol reduced the *Pseudomonas* count well, though not so well as with o-phenylphenol. With 2% and 5% of sodium o-phenylphenol, the *Staph.* count was reduced well, but apparently not so rapidly as with o-phenylphenol.

For comparison, neoprene (G) was tested with no inclusion of bactericide. The *Pseudomonas* count reduced slightly at 24 hours, and the *Staph.* count was greater than 3 million at 24 hours.

EXAMPLE 3

Natural rubber inflations containing 2% to 4% of o-phenylphenol were tested for activity against *E. coli*, saline suspension filling the inflations. The suspension had an initial bacterial count of 304 thousand per ml. A control was maintained in a glass container, and comparison was also made with an untreated natural rubber inflation. The results were tabulated below.

Time, hrs.	Control	Untreated Rubber	Treated Rubber		
			2%	3%	4%
0	304T				
3	460T	230T	51T	68T	20T
9	241T	98T	3.7T	0.1T	0
121	130T	92T	0.2T	0	0

EXAMPLE 4

Inflations constructed of other types of rubber and containing a bactericide were tested for activity. Neoprene (W) containing 5% of o-phenylphenol or 5% of sodium o-phenylphenol reduced the bacterial count of *Staph. aureus* and a *Pseudomonas* species isolated from chicken wattle disease well below a neoprene (W) (an ordinary type of neoprene without sulphur content) control containing no bactericide in 24-hour tests.

In tests against *Staph. aureus*, with an initial bacterial count of 200 thousand per ml., Buna S containing 5% or 6% of o-phenylphenol apparently produced a complete kill and 4% of the agent resulted in a count of 100, in 24 hours. Butyl rubber produced a complete kill in 24 hours at 6% and a

count of 100 at 5% of o-phenylphenol. The control (glass containers) showed increase due to multiplication or cell division.

EXAMPLE 5

Since it is desirable to produce kill as rapidly as possible, preferably within the average time between milkings (10—12 hours), high concentrations of o-phenylphenol were evaluated. It was found that for such articles, the preferred concentration of active agent is 10% to 15%.

As regards the manufacture of the articles, the vulcanization of neoprene was retarded but not excessively at 15% concentration, and the articles had satisfactory properties.

Test results with duplicate neoprene inflations, evaluated as in Example 2, are shown in the table following.

Concentration of agent	Bacterial Count					
	0 hr.	1 hr.	2 hrs.	5 hrs.	6 hrs.	24 hrs.
<i>Staph. aureus</i>						
Glass control (0%)			1200 T		970 T	354 T
Neoprene control (0%)			640 T		150 T	500 T
12%			500 T		35 T	27 T
12%			330 T		35 T	Less than 1000
15%			30 T	less than	10 T	„ 1000
15%			85 T		10 T	„ 1000
¹ 15%			160 T	less than	10 T	„ 1000
² 15%		Less than	60 T	„	10 T	„ 1000
<i>E. coli</i>						
Glass control (0%)			20 T		3340 T	47,000 T
Neoprene control (0%)			40 T		10 T	1000
12%			85 T	Less than	10 T	less than 1000
12%			65 T	„	10 T	„ 1000
15%			20 T	„	10 T	„ 1000
15%			35 T	„	10 T	„ 1000
¹ 15%			15 T	„	10 T	„ 1000
² 15%		less than	10 T	„	10 T	„ 1000

Concentration of agent	Bacterial Count					
	0 hr.	1 hr.	2 hrs.	5 hrs.	6 hrs.	24 hrs.
<i>Pseudomonas</i>						
Glass control (0%)	630 T	735 T		262 T		10 T
Natural rubber control (0%)		620 T		384 T		30 T
Neoprene control (0%)		520 T		420 T		3 T
12%		540 T		154 T	less than	100
12%		450 T		327 T	"	100
² 15%		790 T		20 T	"	100
15%		425 T		65 T	"	100
¹ 15%		690 T		62 T	"	100
15%		785 T		48 T	"	100

¹ Rubber accelerator changed to vary curing.

² Plasticizer omitted for increased hardness.

EXAMPLE 6

A series of four standard types of neoprene milking machine inflations into which had been incorporated from 0.25% to 2% of sodium o-phenylphenol was tested for bactericidal activity. A second series of the inflations was also tested, which contained 0.25% to 2% of di-isobutyl phenoxy ethoxy ethyl dimethyl benzyl ammonium chloride, monohydrate as the bactericidal agent.

Soured milk was allowed to dry on the surface of the inflations. After eight hours, the surfaces of each was swabbed and plated on a nutrient medium to determine the presence of bacteria. No living microorganisms were found.

Simulated field tests with raw milk showed improved results when the inflations containing 15% o-phenylphenol were employed and the inflations were washed with a sterilizing solution after each use, over the use of standard inflations washed in the same manner.

EXAMPLE 7

Field tests were conducted with milking machine inflations on a number of farms over a period of several months, comparing neoprene inflations containing 12% and 15% o-phenylphenol with neoprene and natural rubber inflations containing no bactericidal

agent. Use of the inflations containing bactericidal agent resulted in an average bacterial count of one-half the average count on the controls.

The invention thus provides substantial improvements in handling milk, and a cured microbicidal elastomer article which is effective against the microorganisms found in milk. An especially useful microbicidal milking machine inflation is provided. The invention is very desirable for protecting equipment against bacterial growth due to inadequate sanitation and cleaning methods. The article contains the microbicidal agent therein for effective use over a number of operations.

WHAT WE CLAIM IS:—

1. A cured microbicidal elastomer article employed in contact with milk and having dispersed therethrough at a microbicidally active level a microbicidal agent which is safe for human consumption of the milk at said level.

2. A cured microbicidal elastomer article as claimed in Claim 1, characterized by said agent being a microbicidal phenol.

3. A cured microbicidal elastomer article as claimed in Claim 1 or 2, characterized by said agent comprising orthophenylphenol.

4. A cured microbicidal elastomer article as claimed in Claim 3 characterized by said

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orthophenylphenol being dispersed through the article in a proportion of 0.25% to 20% by weight based upon the raw elastomer content of the article.

5 5. A cured microbicial elastomer article as claimed in any one of Claims 1 to 4, characterized by said elastomer being a synthetic or natural rubber.

10 6. A cured microbicial elastomer article as claimed in any one of Claims 1 to 5, characterized by said article being a milking machine part.

7. A cured microbicial elastomer article as claimed in any one of Claims 1 to 5, characterized by said article being a milking machine inflation. 15

8. A cured microbicial elastomer article substantially as hereindescribed with reference to the accompanying drawing.

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Chartered Patent Agents,
Agents for the Applicants.

Leamington Spa: Printed for Her Majesty's Stationery Office, by the Courier Press.—1961.
Published by The Patent Office, 25, Southampton Buildings, London, W.C.2, from which
copies may be obtained.

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COMPLETE SPECIFICATION

1 SHEET

*This drawing is a reproduction of
the Original on a reduced scale*

FIG. 1

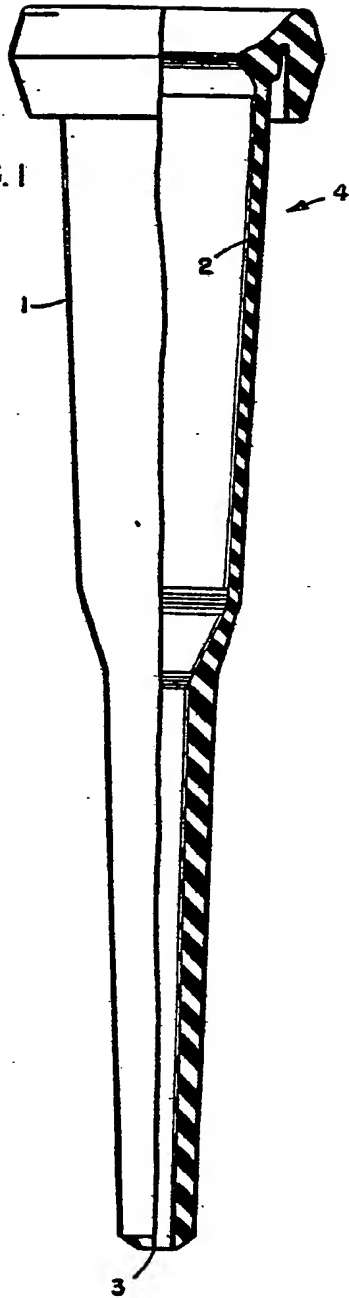


FIG. 2

